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Effect of surface conditioning with air-abrasion on the tensile strength of polymeric CAD/CAM crowns luted with self-adhesive and conventional resin cements

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1. Summary

The aim of this study was to test the tensile strength of polymeric crowns conditioned differently and luted with self-adhesive and conventional resin cements to dentin on the prepared teeth. Human teeth were prepared for all crowns and divided into 10 groups (N = 240, n = 24 per group). Glass ceramic crowns were milled and cemented with dual-polymerized resin cement (Variolink II) (control group) to dentin on the prepared abutments. Polymeric CAD/CAM crowns were milled, and divided into three groups per surface conditioning: (A) no treatment, (B) airborne particle abrasion with 50 μm alumina, (C) airborne particle abrasion with 110 μm alumina, and then luted with one of the following cements: i) RelyX Unicem (Z1, self-adhesive), ii) G-Cem (Z2, self-adhesive), iii) artCem GI (Z3, conventional), iv) Variolink II (C, conventional). The tensile strength was measured initially (n = 12) and after aging by mechanical thermocycling loading (1.200.000 cycles, 49 N, 5-50 °C) (n = 12). Tensile strength (MPa) of all crowns was determined by the pull-off test (Zwick/Roell Z010; Ulm, Germany, 1 mm/min). Data were analyzed with two-way and one-way ANOVA followed by a post-hoc Scheffé test and t-test (p.05). No adhesion of the tested cements was observed on unconditioned polymeric CAD/CAM crowns (0 MPa) and those luted with C type cement. All air-abraded polymeric CAD/CAM crowns (initial: 1.4 - 2.8; 0-2 MPa) showed significantly lower tensile strength values than the glass-ceramic control group (initial: 7.3 MPa; after aging: 6.4 MPa). Among the tested cements, Z2 showed significantly higher values after air-abrasion with 110 pm (initial: 2.8 MPa; after aging: 1 MPa) compared to 50 μm alumina (initial: 1.4 MPa; after aging: 0 MPa). No significant difference was found between 50 and 110 μm particle size alumina with the other two cements. After aging, tensile strength of the crowns luted with Z2 (50 μm: 0 MPa and 110 μm: 1 MPa) and Z3 (50 μm: 1 MPa and 110 μm: 1.2 MPa) were significantly lower than those luted with

Z1 (50 μm: 1.9 MPa and 110 μm: 2 MPa). While with all polymeric specimens failure occurred in the adhesive between the cement and the crowns, the glass ceramic group showed exclusively cohesive failures within the ceramic or in the root of the tooth. Air-abrasion prior to cementation of polymeric CAD/CAM crowns has minimally improved the tensile strength. Both the failure types and the tensile strength values of adhesively luted glass ceramic crowns showed superior results compared to adhesively cemented polymeric ones. Although the tensile strength results were low, crowns cemented with Z1 showed more stable values. The adhesion of polymeric CAD/CAM crowns to dentin was considerably lower than that of the glass ceramic crowns tested.

2. Introduction

The CAD/CAM technology offers the possibility to produce dental restorations by means of numerically controlled machining. This technology has been successfully established for ceramic materials. As an alternative to ceramics, other materials are introduced for dental reconstructions, which can be processed with lower expenditure of time and costs. One such example is polymeric CAD/CAM blocks for temporary dental restorations (All et al. 2011; Stawarczyk et al. 2008; Stawarczyk et al. 2008).

Since these CAD/CAM blocks are industrially polymerized under high pressure and temperature, they present higher mechanical properties compared to the manually polymerized resins (Alt et al. 2011; Balkenhol et al. 2008; Basaran et al. 2011, Stawarczyk et al. 2008; Stawarczyk et al. 2008; Stawarczyk et al. 2009). In general, while the manually polymerized resins show inferior fracture resistance, they are only indicated for interim reconstructions (Alt et al. 2011; Balkenhol et al. 2008; Basaran et al. 2011, Stawarczyk et al. 2008; Stawarczyk et al. 2008; Stawarczyk et al. 2009). Due to their good optical and mechanical properties, as well as their less abrasive effect on the antagonist enamel, recently introduced polymeric CAD/CAM blocks are considered as alternative materials to glass ceramics (Ghazal and Kern. 2010; Fischer et al. 2008). However, limited information is available on their mechanical durability with and without aging regimens. One study reported that after 3 months of water storage at 37°C and 5000 thermocycles, industrially polymerized 3-unit FDPs showed significantly higher fracture load compared to manually polymerized ones (Alt et al. 2011). Another study observed no impact of saliva storage for 180 days of CAD/CAM resins (Stawarczyk et al. 2009).

Since these materials are also indicated for long-term restorations, their adhesion is of importance for their durability. To the authors' best knowledge, at present, there is no information available on the retentive strength of polymeric CAD/CAM crowns. Adhesion of resin-based cements includes both conditioning the cementation surface of the restorations as well as the prepared dentin. One of the most common methods to condition polymeric materials is the use of sandblasting, which in principle cleans the surface and at the same time increases the surface area (Ersu et al., 2009; Marshall et al. 2010). Similar effects are observed in glass ceramics after hydrofluoric acid etching (Naves et al. 2010).

Adhesion has two aspects, and for durable restorations not only the conditioning of the restorative material but also the dentin is crucial for good attachment of the resin cement to both substrates. Etching-and-rinse bonding systems are still considered the gold standard for conditioning of dentin. However, due to their technique sensitivity, some of the conventional resin cement systems involve self-etch adhesives. On the other hand, self-adhesive cements do not require any conditioning of the dentin, which eliminates technique sensitivity (Behr et al. 2004).

Adhesion of such cements could be individually tested either on the restoration material or on the tooth substrate (Oilo, 1993; Mesquita et al., 2010). However, in order to simulate the clinical environment under more realistic conditions, investigation of the tensile strength of luting agents can be studied using a pull-off test with axial dislodgement forces, with crowns luted to extracted human teeth (Ernst et al. 1998).

In addition, with pull-off tests other conclusions can be taken (Ernst et al. 2007) in comparison with tensile tests like μTBS (Escribano et al. 2006). The pull-off testing procedure is complex and technique-sensitive but provides information on the retentive performance of a material (Ernst 1998).

This study tested the impact of sandblasting using two particle sizes and different resin cements on the tensile strength of polymeric CAD/CAM crowns bonded to dentin. The hypothesis tested were a) whether the tensile strength of polymeric crowns would be similar to glass ceramic crowns, b) sandblasting would increase the tensile strength.

3. Materials and Methods

This study tested the tensile strength of sandblasted or non-treated polymeric CAD/CAM crowns luted with two self-adhesive resin cements (RelyX Unicem, Z1; and G-Cem, Z2) and conventional resin cement (artCem Gi, Z3). Glass-ceramic crowns bonded using conventional resin cement (Variolink II, C) were employed as control group. Table 1 describes the tested groups.

3.1 Specimen preparation

Extracted caries-free molars (N = 240) were collected and cleaned from periodontal tissue residues, stored in 0.5% Chloramine T at room temperature for a maximum of 7 days. Thereafter, they were stored in distilled water at 5°C for a maximum of six months according to ISO/TS 11405:2003. Before the teeth were embedded, they required pre-treatment. The roots were shortened to a length of 10mm, and retention holes with a depth of approximately 1mm were prepared in the roots. Then, each tooth was embedded in a special holding device parallel to the tooth axis using acrylic resin (ScandiQuick, SCAN DIA, Hagen, Germany).

The teeth were prepared with a motorized parallelometer (PFG 100, Cendres Métaux, Biel-Bienne, Switzerland) with a conicity of 10° (Figure 1 a) and shoulder preparation with 40 μm using a diamond coated dental bur (FG 305L/6, Intensiv SA, Grancia, Switzerland). In order to get a standardized coronal height of 3 mm, the holding device was positioned in a cut-off grinding machine (Accutom-50, Struers GmbH, Ballerup, Denmark). With a polishing disc (Sof-Lex 1982C/1982M, 3M ESPE; Seefeld, Germany) the edges of the coronal part were rounded. The specimens were stored in water at 37°C prior to cementation and testing.

The prepared abutments were scanned with a Cerec 3D camera (Sirona, Bensheim, Germany), and the bond surface area was calculated (Cerec Software 2.80 R2400 Volume Difference, Sirona). For each abutment, the crowns were designed (Cerec InLab 3D Program Version 3.10, Sirona) and milled with Cerec InLab XL (Sirona).

Glass ceramic crowns (VITA Mark II, VITA Zahnfabrik, Bad Säckingen, Germany) were etched (9% buffered hydrofluoric acid: Ultradent Products, South Jordan, USA) and treated with a silane couping agent (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein) and an adhesive (Heliobond, Ivoclar Vivadent) according to the manufacturer's instructions. The teeth surfaces were conditioned with Syntac Classic (Ivoclar Vivadent) and crowns were cemented with resin cement (Variolink II, Ivoclar Vivadent) according to the manufacturer's instructions.

The CAD/CAM crowns were conditioned as follows (N=24 for each pre-treatment group and adhesive resin cement):

Group a: no treatment.

Group b: sandblasted with Al_20_3 50 μm for 10 s at a pressure of 2 bar from a distance of 10 mm

Group c: sandblasted with Al_20_3 110 μm for 10 s at a pressure of 2 bar from a distance of 10 mm

Then, the CAD/CAM resin crowns were bonded according to the manufacturers' instructions on dentin prepared teeth (Table 1). The cements were light-polymerized for 30 s (Elipar S10, 3M ESPE). Subsequently, the specimens in all groups were stored in an incubator for 10 min at 37°C and loaded in a special device with 100 N.

2.2. Aging of specimens

While the initial tensile strength was measured in half of each group (n=12), the other half (n=12) was subjected to mechanical thermocycling loading (chewing simulator, University of Zurich). The crowns were mechanically loaded with 49 N for 1.2 million times at a frequency of 1.67 Hz. Simultaneous thermocycling was achieved by changing the surrounding water temperature in the chamber every 120 s from 5°C to 50°C. Mesiobuccal cups from nearly identical upper human molars fixed in amalgam acted as antagonists. This buffer is bonded to the socked with an angle of 15 degree. The function of this attachment is to act as desmodont (Rateitschak et al. 1978). The No Aging groups did not undergo mechanical thermocycling loading.

2.3 Tensile strength test

To embed the crowns in the upper holding devices and position the lower holding devices parallel with 1.5 mm space between each other, the space between the lower holding devices was filled with an additional silicone (Lab Putty: Coltène/Whaledent, Altstätten, Switzerland). Acrylic resin (ScandiQuick) was poured through the screw hole in the bottom of the holding device.

To obtain the tensile strength, the specimens were pulled off (Universal Testing Machine, Zwick/Roell Z010: Zwick, Ulm, Germany) at a cross-head speed of 1mm/min until the two holding devices disconnected and debonding took place (Fig. 1). The tensile strength of specimens that failed before actual testing was considered to be 0 MPa. The bond strength values (N/mm^2 = MPa) were then calculated as the ratio between the tensile strength and the bond area (as measured using Cerec Volume Program, Sirona).

2.4 Failure types

After tensile strength testing, the failure types were classified in three main groups: i) failure in the interface of dentin and cement, ii) mixed failure and iii) failure in the interface of polymeric crown and cement. For the failure type classification, an optical microscope at a magnification of 25 times was used and pictures were made

(Tesovar: Zeiss, Zurich, Switzerland) to collect more detailed information on the observed failure types.

2.5 Statistical analysis

Tensile strength data were analysed using descriptive, one-way ANOVA followed by the Scheffé test and the Student's t-test. P-values smaller than 5% were considered to be statistically significant in all tests. The statistical analysis was made using Statistical Package for the Social Science Version 15 (SPSS Inc., Chicago, IL, USA).

4. Results

The adhesively luted glass ceramic crowns (control group, C) showed the highest tensile strength compared to all other test groups before and after mechanical thermocycling loading (Table 2, Fig. 2). Aging just slightly influenced the results in the control group (Table 3).

The non-conditioned polymeric crowns with all cement groups showed no bonding (Table 2, Fig. 2).

Except for sandblasting (50 μm) and the aged Z2 cement group, where all specimens were debonded after mechanical thermocycling loading, all other sandblasted groups showed significantly higher results compared with non-treated groups (Table 4).

The Z2 cement in both initial and aged group, sandblasted with 110 μm alumina, showed higher tensile strength results compared to sandblasting with 50 μm alumina. Within the 50 μm alumina sandblasted groups, Z2 cement showed the lowest initial tensile strength compared to those of other groups.

No significant differences were found with sandblasting using 100 μm alumina among the initial test groups. After aging, the tensile strength of Z1 cement was significantly higher than that of Z2.

During the measurement of tensile strength, the glass ceramic crowns were fractured cohesively (Fig. 3C) or in the root (Fig 3D). CAD/CAM resin crowns were fractured adhesively between the cements and the crowns (Fig. 3B) or in the interface dentin/cement and crown/cement (Fig. 3A).

5. Discussion

Non-treated CAD/CAM resin crowns showed no bonding for all cements. Pre-treatment with alumina improved the results. The tensile strength of pre-treated resin crowns cemented with all tested cements presented significantly lower values than those of the adhesively luted glass ceramic crowns (control group). Consequently, the first hypothesis was rejected.

The control group showed the highest tensile strength compared to all resin groups. The glass ceramic crowns were cohesively fractured in both initial and aged group. Consequently, the measured tensile strength of adhesion exceeded the cohesive strength of the ceramic or the root (Figure 3). Therefore, this test method could not be adapted for glass ceramic crowns due to the lower flexural strength of the ceramic tested. This phenomenon was also observed with other test methods such as shear bond strength test where cohesive is often found in the glass ceramic (Mesquita et al. 2010).

The second hypothesis tested the influence of sandblasting on the tensile strength of CAD/CAM resin crowns compared with non-treated ones. The sandblasted crowns presented higher tensile strength at all times. Therefore, this hypothesis could be accepted (Table 2).

Sandblasting principally cleans and increases the surface area resulting in higher bond strength due to mechanical retention (Ersu et al. 2009; Marshall et al. 2010). Based on the results of this study, the adhesion between the polymeric crowns and the resin cements could be considered as mechanical retention. The polymeric blocks are industrially polymerized and have a high degree of conversion compared to manually polymerized ones (Pereira et al. 2010). Since the non-treated group showed no bonding, it can be stated that free radicals were not sufficient to achieve adhesion between the studied cements and the intaglio surfaces of the crowns.

This study used the pull-off test using prepared human teeth, where CAD/CAM resin crowns were bonded according to standard clinical procedures (Table 1). However, the teeth were prepared manually, and the water supply was not controlled with the handpiece as under clinical conditions. In a previous study, where tensile strength of zirconia crowns cemented with self-adhesive resin cements on dentin were tested (Stawarczyk et al. 2011), the results ranged between 7.3 MPa and 14.1 MPa. Although the identical experimental set-up was used, the results of this study indicated inferior adhesion of two of the cements (Z1, Z2) on the polymeric crowns.

The benefit of pull-off tests is the integration of the surface bonded area in the calculation. It can be assumed that the applied method presents a more precise calculation than previously published studies (Ernst et al. 2005; Ernst et al. 1998; Palacios et al. 2006; Yim et al. 2000). In other studies, bond area was measured by wrapping 0.1 mm tinfoil around the preparation to determine the weight of the foil (Ernst et a. 2005; Ernst et al. 1998). In two other studies, bond area of the specimens was calculated using the formula for a truncated cone to which the area of the flat occlusal surface was added (Palacios et al. 2006; Yim et al. 2000). Only in our studies in Zurich, the prepared abutments were scanned with a Cerec 3D camera and the areas were calculated with the Cerec 3 Volume Program (Stawarczyk et al. 2011).

In this study, the specimens were additionally subjected to chewing simulation, where the stress for all specimens was standardized and reproducible. The use of a specially developed chewing simulator with additional thermocycling is a well-established method to simulate the clinical situation (Manhart et al. 2001; Göhring et al. 2003; Stawarczyk et al. 2011). Mechanical loading of 1.200.000 cycles was claimed to correspond to 5 years in vivo (Lutz and Krejci, 1994). Subsequently, the specimens in this study were aged with the equivalent of 5 years of in vivo use.

In summary, the CAD/CAM resin crowns showed significantly lower tensile strength than the control group. Further studies should also test other pre-treatment methods for industrially polymerized resins such as silanization, silica-coating or application of methacrylate monomers.

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7.Tables and Figures:

Table 1: Summary of resin cements, abbreviations, bonding agents, their composition and application

Composition of used materials			
		Composition	**Application**
Cement	**Abbreviation**		
Variolink II (Lot.No K41833/K39878) VITA Mark II, VITA Zahnfabrik, Bad Säckingen, Germany	C	Bis-GMA, TEGDMA, UDMA benzoylperoxide, anorganic fillers, ytterbium trifluoride, Ba-Al fluorosilicate glass, spheroid mixed oxide, initiator, stabilizers, pigments Syntac Classic (Lot.No J280035/J27820) Monobond S (Lot.No J17658) Heliobond (Lot.No G09457) All: Ivoclar Vivadent, Schaan, Liechtenstein	1. Enamel, dentin etching 2. Mix Variolink II in a 1:1 ratio on a mixing pad for 10s. 3. Placement and light cure each surface for 40s. Primer: TEGDMA, maleic acid. Dimethacrylate, water Adhesive: PEGDMA, maleic acid, Glutaraldehyde, water Ethanol, water, silane Bis-GMA, dimethacrylate, initiators, stabilizers
RelyX Unicem Aplicap (Lot.No 361930) 3M ESPE, Seefeld, Germany	Z1	Powder: alkaline (basic) fillers, silanated fillers, peroxy components, pigments, substituted pyrimidine Liquid: methacrylate monomers containing phosphoric acid groups, acetate initiator components, stabilizers	1. Insert capsule into activator, press handle and hold for 2-4s 2. Mix 10s with RotoMix Capsule Mixing Unit and apply cement to the prepared restoration. 3. Place the restoration and remove excess. 4. Light cure for 2-4s, remove excess. 5. Light cure each surface for 40s.
G-Cem (Lot.No 0801091) GC Europe, Leuven, Belgium	Z2	Powder: fluoro-alumino-silicate glass, Initiator, pigments Liquid: 4 – META, UDMA, aluminio-silicate glass, dimethacrylate, water, phosphoric ester monomer, initiator, camphorquinone	1. Shake or tab to loosen powder, depress plunger. 2. Click once in capsule applier to activate, mix with RotoMix for 10s. 3. Place the restoration and remove excess. 4. Light cure for 2-4s, remove excess. 5. Light cure each surface for 40s.
artCem GI (Lot.No 7806520) Merz Dental	Z3	Powder: barium-aluminium-silicate glass, nano-fluorapatite, pigments, Photo initiator Liquid: poly acid, metaclyate, initiator,	1. Shake or tab to loosen powder, depress plunger. 2. Press Capsule as deep as possible in the activator to activate it. 3. Mix 15s with RotoMix Capsule Mixing Unit and apply cement to the prepared restoration. 4. Light cure for 2-4s, remove excess. 5. Light cure each surface for 40s.
Adhesive artCem ONE (Lot.No 5811037)		2-hydroxyethylmethacrylate, dimethacrylate, initiator, stabilizers	1. Apply evenly on the tooth, waiting fpr 20s 2. Blow smoothly for 10s 3. Polymerization for 20s

Table 2: Tensile strength values (MPa) of all tested groups with minimum, median and maximum tensile strength.

Groups	Pre-treatment	Aging	Mean (SD)	Min	Median	Max
C	etched	Initial	7.3 (2.2)			
	etched	Aging	6.4 (0.9)			
Z1	No treatment	Initial	0 (0)	0	0	0
		Aging	0 (0)	0	0	0
	50µm Al_2O_3	Initial	2.2 (0.15)	1.5	2.2	3.1
		Aging	1.9 (0.20)	0.7	1.9	3.1
	110µm Al_2O_3	Initial	2.6 (0.28)	1.0	2.8	4.0
		Aging	2.0 (0.33)	0.7	1.6	4.4
Z2	No treatment	Initial	0 (0)	0	0	0
		Aging	0 (0)	0	0	0
	50µm Al_2O_3	Initial	1.4 (0.22)	0.5	1.1	2.9
		Aging	0.0 (0.0)	0.0	0.0	0.3
	110µm Al_2O_3	Initial	2.8 (0.15)	2.1	2.7	3.9
		Aging	1.0 (0.20)	0.4	0.7	2.6
Z3	No treatment	Initial	0 (0)	0	0	0
		Aging	0 (0)	0	0	0
	50µm Al_2O_3	Initial	2.1 (0.13)	1.6	2.0	3.0
		Aging	1.0 (0.19)	0.2	0.8	2.0
	110µm Al_2O_3	Initial	2.3 (0.15)	1.5	2.4	3.2
		Aging	1.2 (0.13)	0.5	1.0	2.2

Table 3: P-values of the two sample Student's t-test with mean difference and 95% confidence interval between initial and aging groups within one pre-treatment and within each cement.

Group	Pre-treatment	p-value	Mean difference
C	Etched	0.416	0.83
Z1	No treatment	**-**	**-**
	50µm Al_2O_3	0.231	0.31
	100µm Al_2O_3	0.151	0.65
Z2	No treatment	**-**	**-**
	50µm Al_2O_3	**<0.001**	1.37
	100µm Al_2O_3	**<0.001**	1.82
Z3	No treatment	**-**	**-**
	50µm Al_2O_3	**<0.001**	1.16
	100µm Al_2O_3	**<0.001**	1.15

Table 4: P-value of the two sample Student's t-test with mean difference and 95% confidence interval between with 50 µm Al_2O_3 and 100 µm Al_2O_3 sandblasted groups within aging or initial and within each cement.

Group	Aging / No Aging	p-value	Mean difference
Z1	Initial	0.230	-0.39
	Aging	0.932	-0.03
Z2	Initial	**<0.001**	-1.44
	Aging	**<0.001**	-0.99
Z3	Initial	0.378	-0.18
	Aging	0.421	-0.19

Figure 1 A: Motorized parallelometer with conicity of 10°. **B:** Design of tensile bond strength measurement

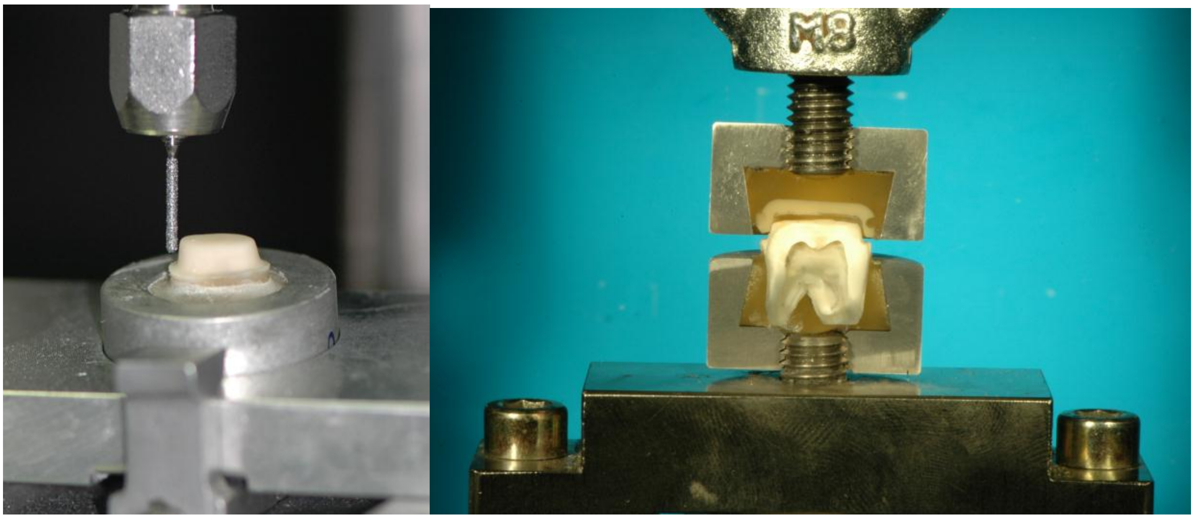


Figure 2: Mean tensile strength results of all tested groups.

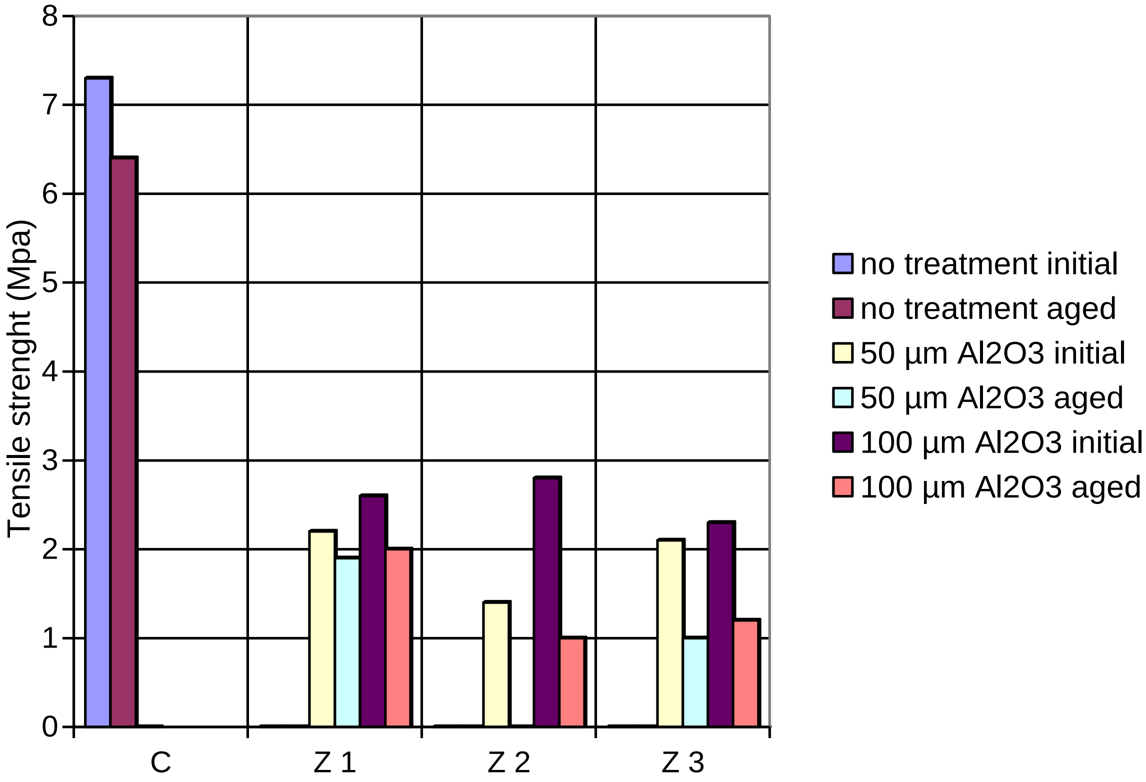
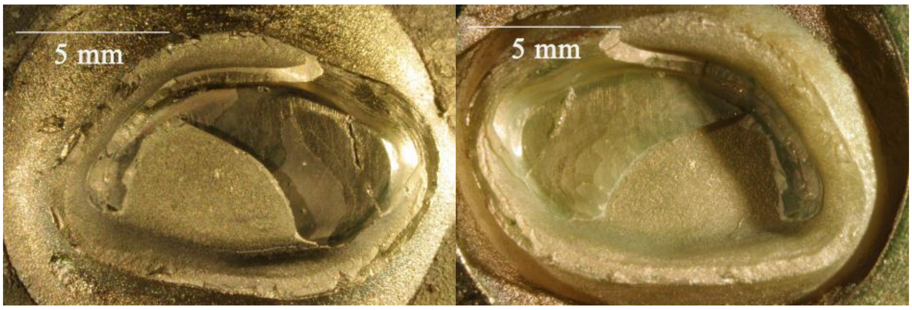
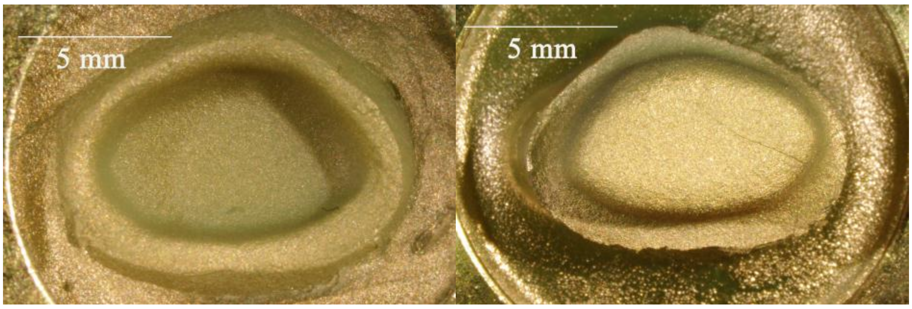


Figure 3: Failure types after tensile strength measurements.

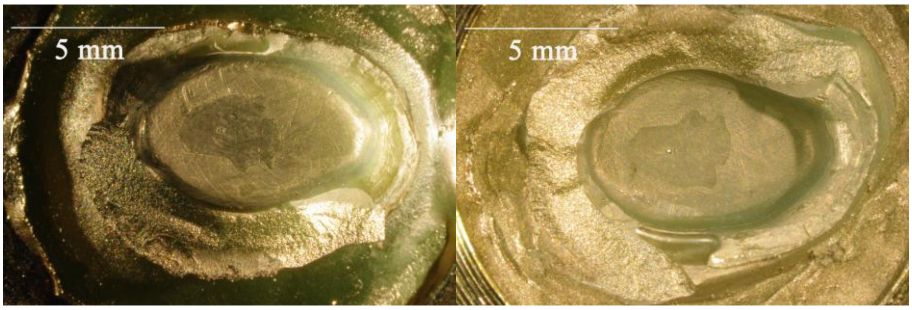
A: Fracture in interface dentin/cement and crown/cement



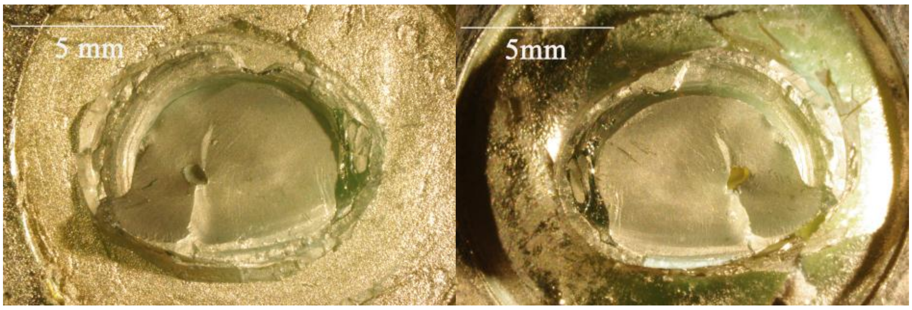
B: Fracture in the interface crown/cement



C: Fracture of glass ceramic crown



D: Fracture of a root of a glass ceramic crown



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